Which metallic alloys can be used as pressure cell windows? The case of SANS

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Task 2: "Kinetics and Dynamics"

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Summary

✓ Aim of the study :

-> Pressure cell for SANS and NSE measurements, up to 6,000 bar, with small volumes (≈ 100 µL) and low concentration (typically few g/L for a protein) to study structure and dynamics of molecules in soft matter and biophysics (*e.g.* conformation of proteins, ...)

✓ Our strategy:

* thick **windows** in stainless materials (**alloys**), which display good mechanical properties, reasonable transmissions, and (if possible...) a « low » Q-scattering

* use of a method developped at LLB for **cell subtraction**:

- A. Brûlet *et al. J Applied Crystallography* 40: 165-177, 2007
- « Pasinet » reduction software: http://didier.lairez.fr/pasinet2/doku.php

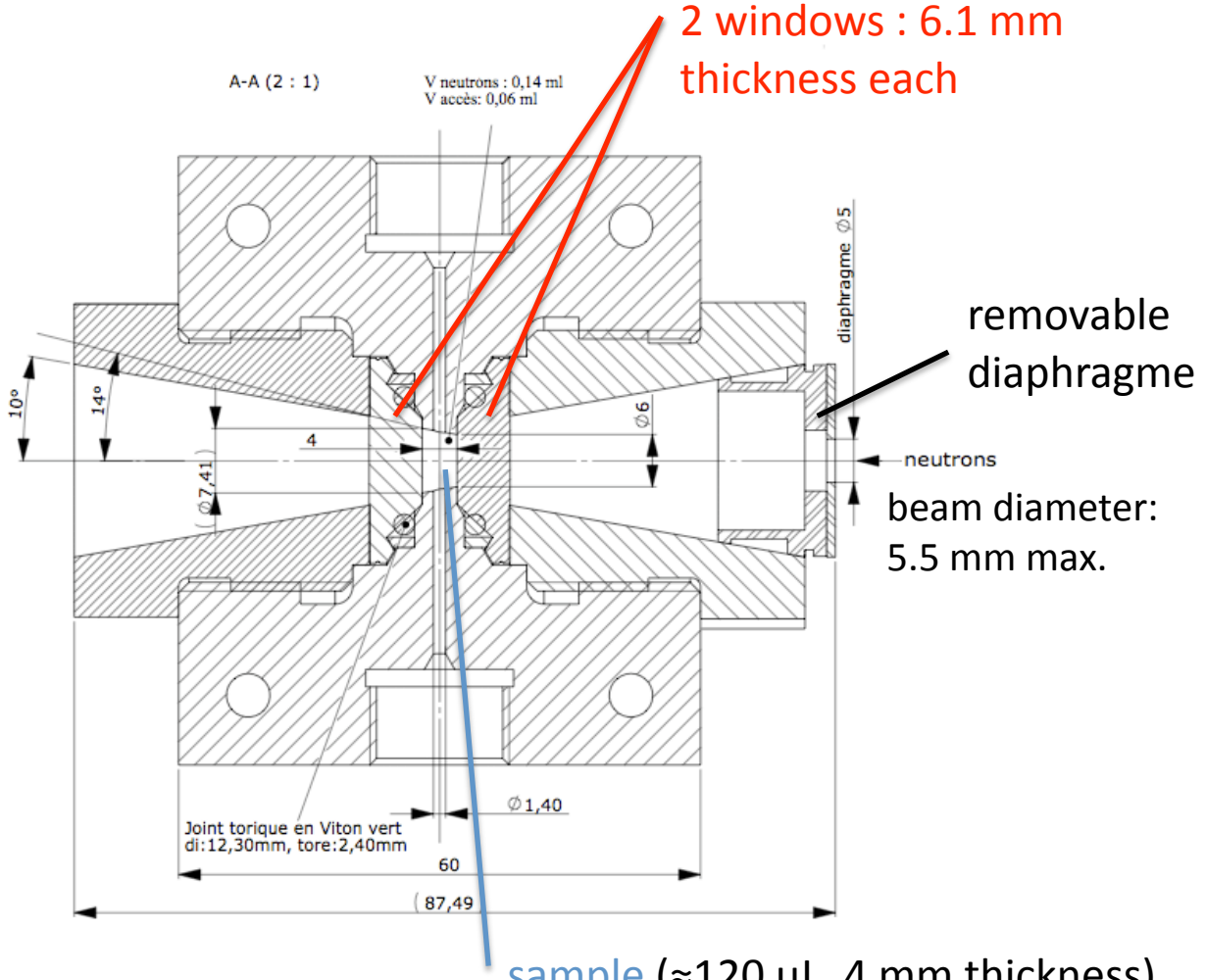
Pressure cell with removable windows

✓ cell body: Marval x12 stainless and extremely resistant → reusable

✓ windows: cheap (≈ 100 €) and easy to machine
→can be changed
→ adaptable to the experiment

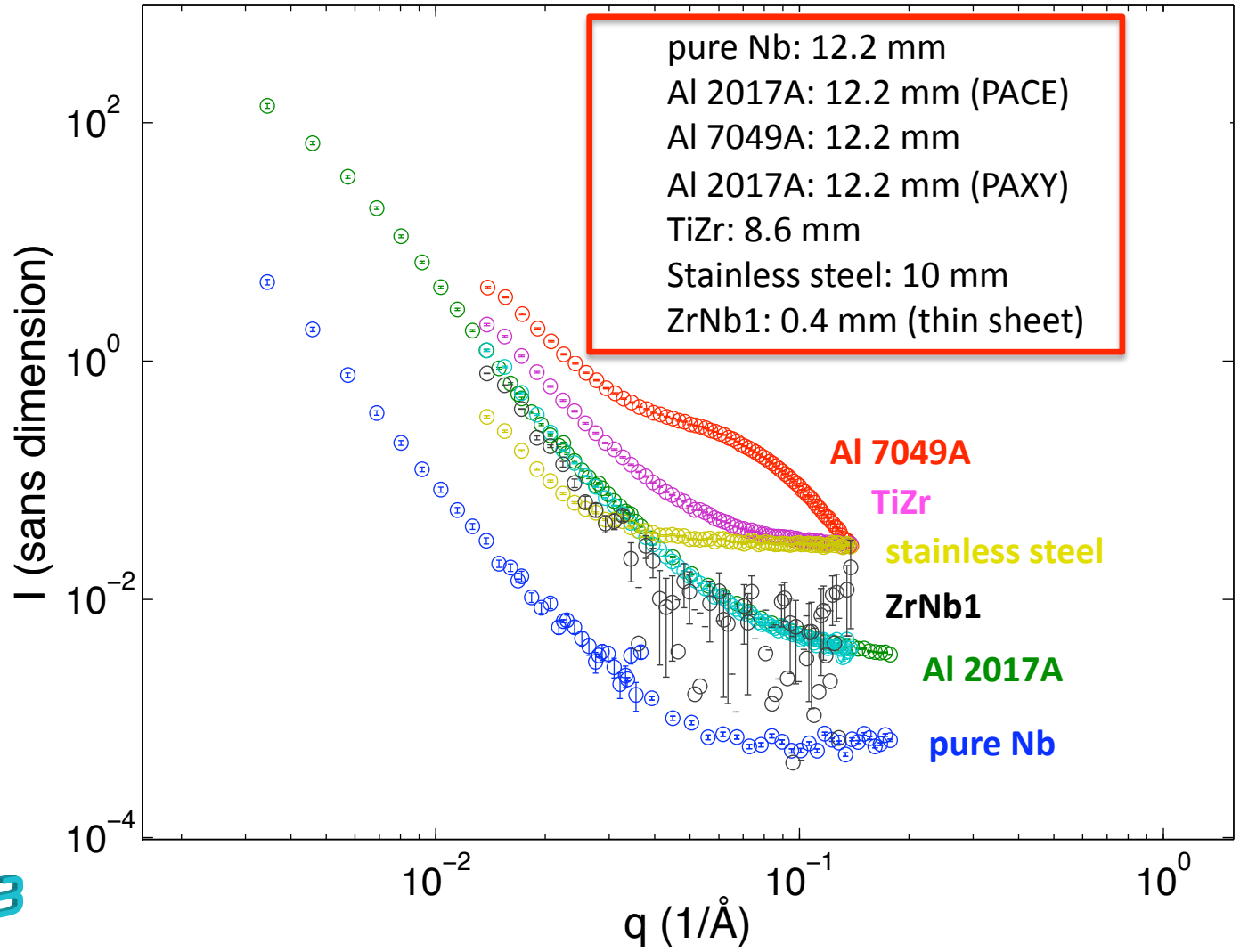
✓easy to clean and empty

✓ temperature control (15-140°C)

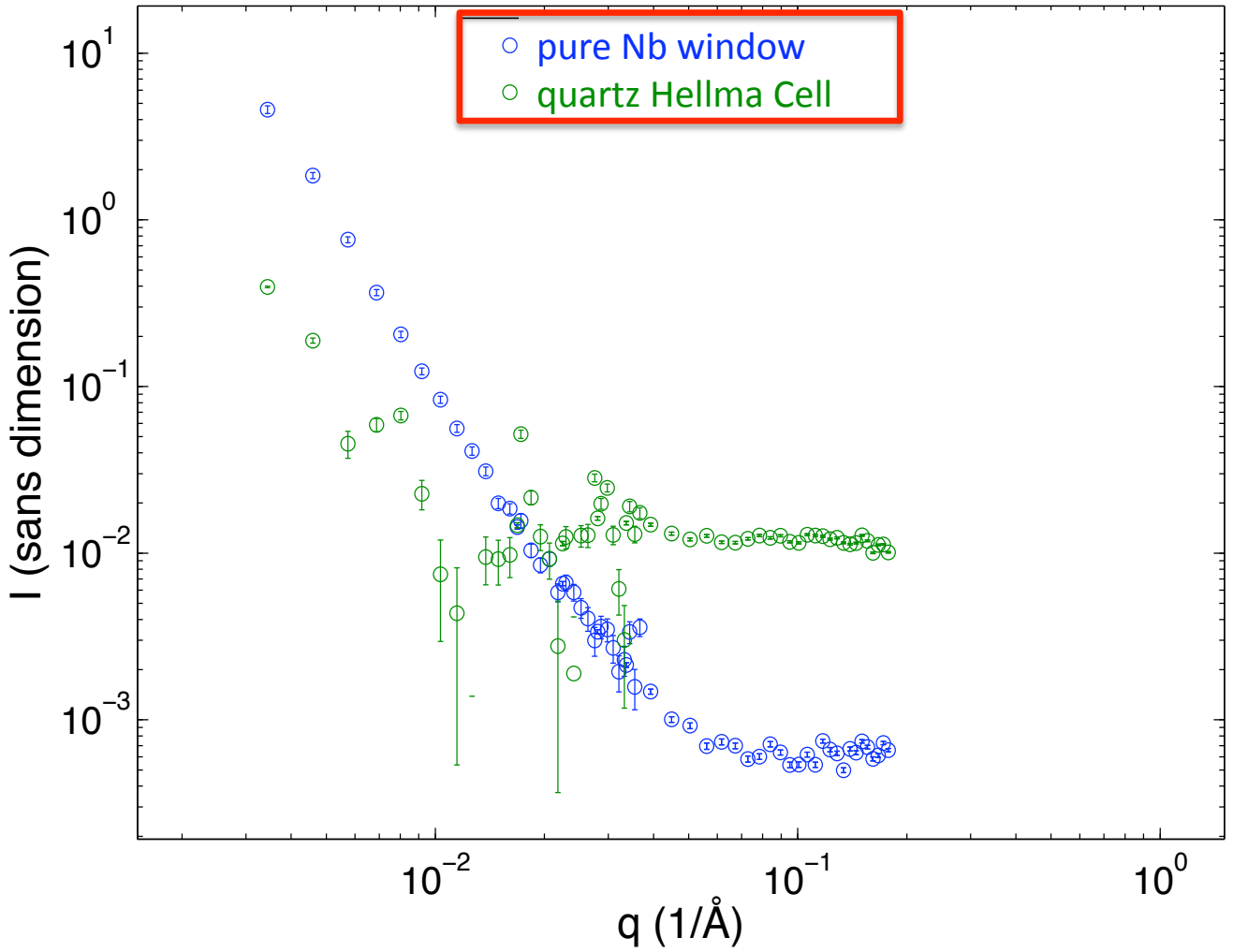


Nb *vs.* other alloys

Window thickness for P_{max} ≈ 6,000 bar:
6 Å-3 m : 0.01 < Q < 0.15 $Å^{-1}$, **normalization by the thickness**



Nb *vs.* quartz Hellma cell



Which alloy?
Tr, P_{max}

	Total thickness (mm)	Transmission at 13 Å	**Tr at 13 Å for 10 mm thickness**	Transmission at 6 Å	**Tr at 6 Å for 10 mm thickness**	Max. pressure (bar) **for 10 mm**	Q range ($Å^{-1}$)
Al 2017A	12.2	0.785	**0.820**	0.948	**0.957**	~4500	Q > 0.01
Alu 2017A + Mb	12.2 + Mb	0.506		0.556			
Al 7049A	12.2	0.760	**0.799**			~6000	Q > 0.1
	24.4			0.884	**0.951**		
Alu 7049A + Mb	12.2 + Mb	0.497		0.544			
~~**TiZr** (52.5-47.5)~~	8.6			0.416	**0.361**	~6000	Q > 0.07
TiZr + Mb	8.6 + Mb			0.261			
~~**Stainless steel**~~	10			0.295	**0.295**	~6000	Q > 0.01
ZrNb1	0.4			0.993	**0.839**	~3000	Q > 0.01
pure Nb	12.2	0.595		0.775	**0.811**	~2500	Q > 0.01
Quartz Suprasil	2.5	0.970	**0.885**	0.950	**0.815**	-	Q > 10^{-3}
Saphir *	12	0.850 (18 Å)	**0.873** (18 Å)	0.940	**0.950**		

* from Ralf Schweins

Alloy window tests: conclusions

✓ Good subtraction of the pressure cell windows (Al, Nb, TiZr)

✓ **Pure Niobium** (from Cabot, Boyertown (USA)): soft metal ; difficult to machine but resistant to corrosion
but a good candidate for low scattering samples (*e.g.* diluted proteins), at low pressure experiments (P < 2,5000-3,000 bar)

✓ Removable windows: adaptable to the experiment

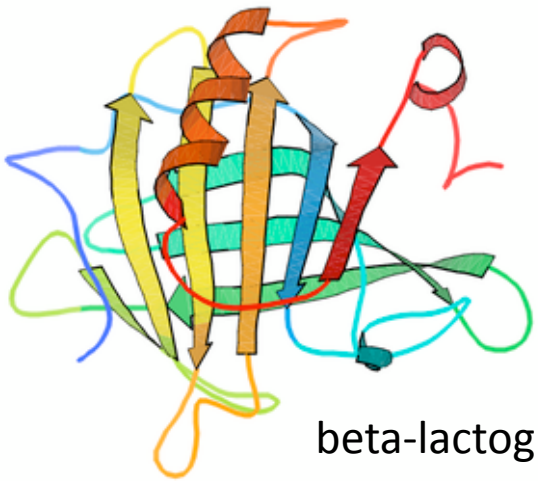
- range of Q-scattering
- absorption
- P_{max}
- corrosion

A real experiment with the pressure cell

Aim of the study: investigate the conformations adopted by the beta-lactoglobulin (BLG) (milk protein) under pressure in the presence of retinol (A vitamine)

→ determine if the presence of this small molecule (retinol) inside the protein stabilizes its structure.

BLG is very sensitive to high pressure, whereas myoglobin, with its compact structure, is less affected by pressure. Unfolding of beta-lactoglobulin begins around 2'500 bar, while it is observed at 6,000 bar in the case of myoglobin.

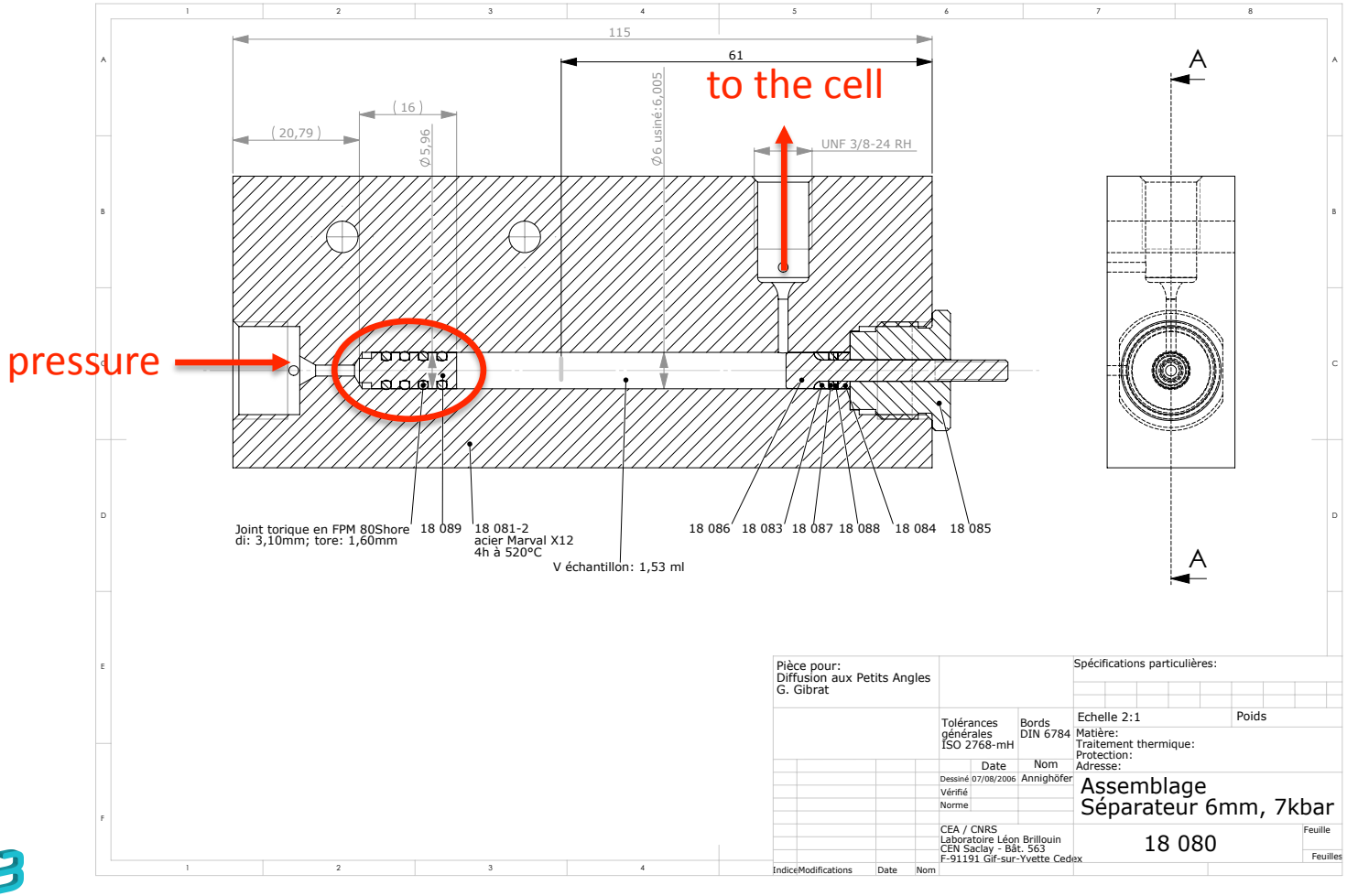


beta-lactoglobulin

different concentrations: 5, 10, 15 g/L
(higher: aggregation)

Results

✓ **Separation piston**: good sealing between the buffer in the sample room → no problem of sample or buffer leakage or mixing of sample/buffer solutions



Difficulties

✓ **Leakage**: the Nb windows had been tested until 2,700 bar ; but during the experiment, leakage at 2,200 bar ; measurement done at 1,800 bar, not enough to unfold the BLG protein

✓ **Change of the windows**: (Nb -> Al 2017A) quite long because of (soft and malleable) **lead** O-ring (more than 1h) and tricky (the cell could be damaged) ; has to be done before the experiment, not during a 3-day-test!

→ several replacement cells (but expensive: about 4,000 €/cell)

→ change of the system of sealing, without using lead O-ring

Solution?

Bridgman system:
over pressure, use of **plastic seals instead of lead!**

other system:
leakage possible? but easy to demount

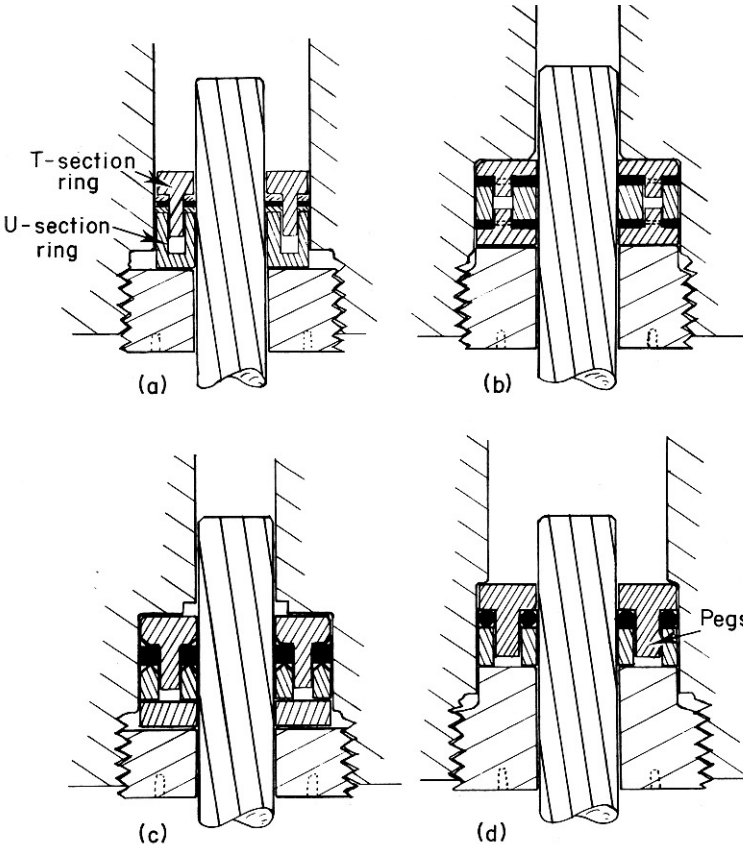


Figure 5.32 Mushroom-type gland seals: (a) after Griggs (1936), (b) after Tsikl (c) after Lifshits and Martinov (1963), and (d) the 'Dowty' seal (unsupported ar from the holes through which the pegs pass)

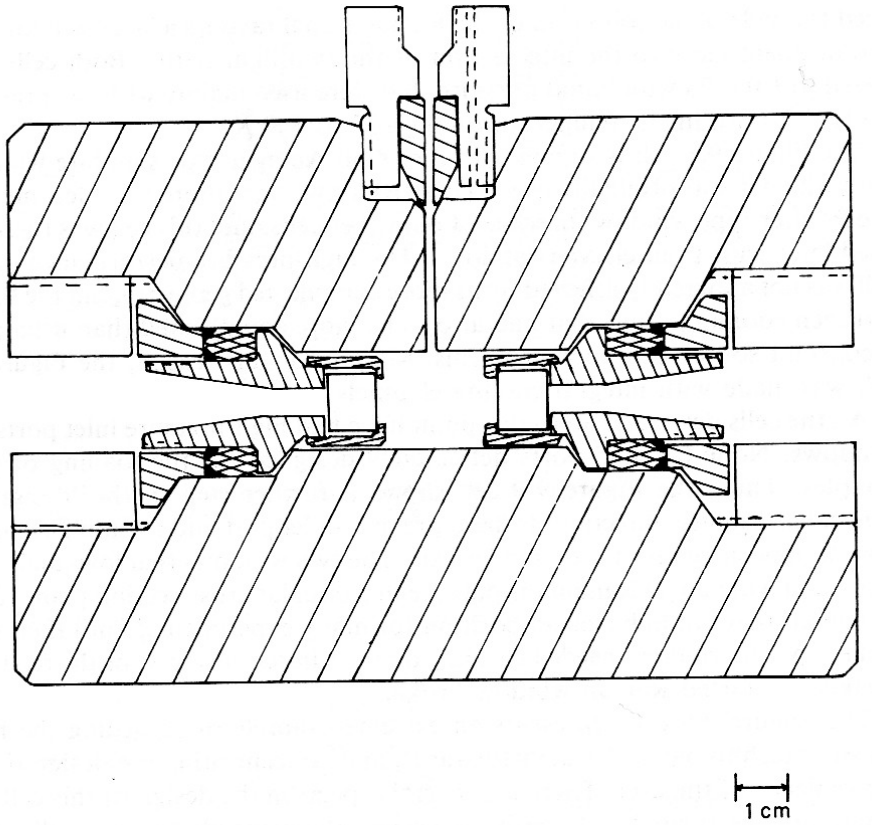
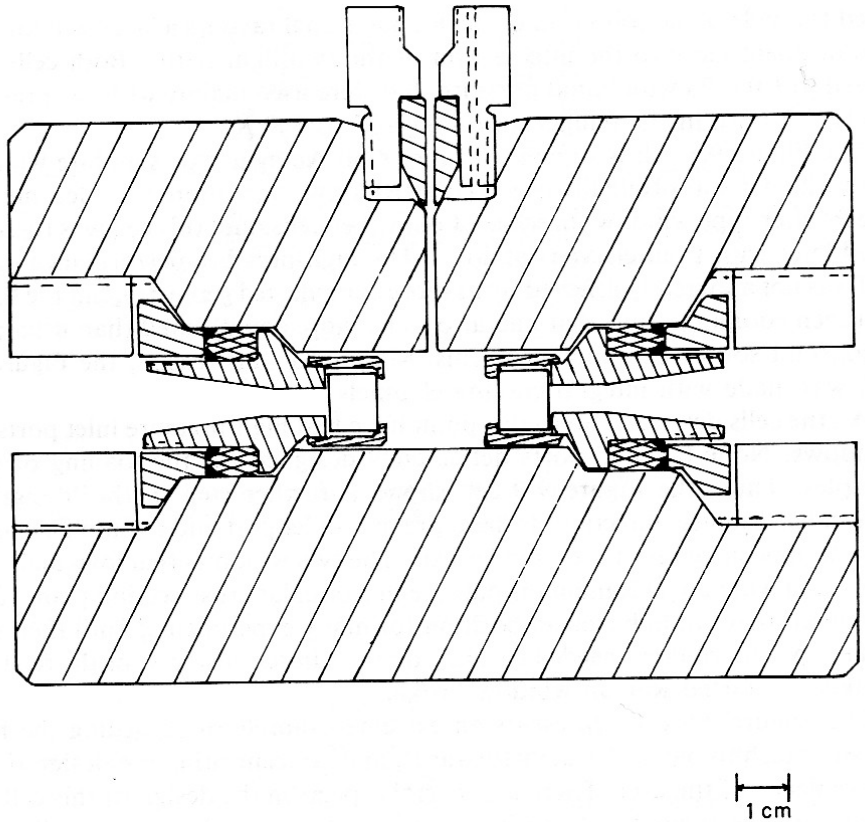
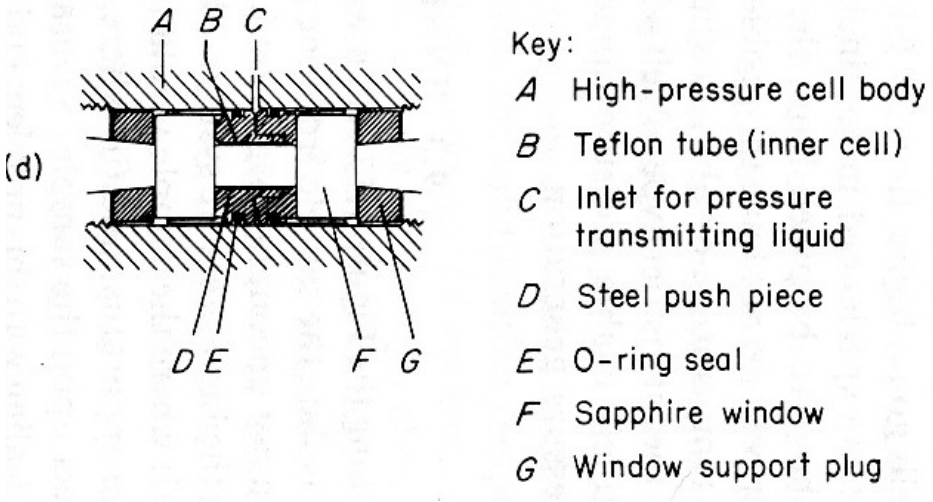


Figure 9.10 Two-window steel cell after Dultz, Krause and Ploner (page 442, Vol. 1. *Proc. VI AIRAPT Conf.* (1977); Plenum Press, New York (1979)) for Raman studies (forward or back scattering) $0 \leqslant P \leqslant 1$ GPa, $70 \leqslant T \leqslant 600$ K

W.F. Sherman, A.A. Stadtmuller, Experimental techniques in high pressure research, John Wiley & Son LTD, ISBN 0 471 10313 6 (1987)

Solution?





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